

HIGH TEMPERATURE THERMOCOUPLE
RESEARCH AND DEVELOPMENT PROGRAM

MONTHLY PROGRESS REPORT NUMBER 2
Period 1 July 1963 to 1 August 1963
Contract Number NAS 8-5438
Request Number TP 3-83547

prepared for
GEORGE C. MARSHALL SPACE FLIGHT CENTER
Huntsville, Alabama

work performed by
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5251 West Imperial Highway
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Date of Publication: 10 August 1963

FACILITY FORM 602

N66-87542	(THRU)
(ACCESSION NUMBER)	<i>None</i>
29	(CODE)
(PAGES)	
CAC 78784	(CATEGORY)
(NASA CR OR TMX OR AD NUMBER)	

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ABSTRACT

This report covers the period 1 July 1963 to 1 August 1963, under Contract NAS 8-5438, which calls for twelve months of research and development of a high temperature thermocouple capable of measuring rocket engine exhaust temperatures in the 3000°C temperature range, under adverse conditions of oxidation erosion, vibration, and shock.

The primary objectives of the program are to advance the state of the art of high temperature thermometry, and to develop an end product suitable for in-flight temperature measurements on the SATURN vehicle.

Preliminary investigations of design parameters led to the selection of a basic configuration for the first three prototype gauges to be delivered under the contract. Shipment of these gauges is now scheduled for 17 October 1963.

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SECTION I

SUMMARY

1.0 Period Covered

This report covers the period 1 July 1963 to 1 August 1963.

1.1 Statement of Work

The Contractor shall advance the state-of-the-art of high temperature thermometry and specifically improve the technique of accurately measuring high temperatures by designing, fabricating, testing, and delivering nine (9) thermocouple probes capable of operation in the 3000°C temperature range under adverse conditions of erosion, oxidations and high stress levels for useful periods of time. Also, present methods of thermocouple probe fabrication will be modified such that the end product will be suitable for in-flight temperature measurements on the SATURN vehicle.

To accomplish the above objectives, the Contractor shall consider and explore specific R&D efforts as follows:

- a. Development of the physical structure of an immersed probe to attain minimum drag and highest resistance to bending and shear forces.
- b. Ascertain the best combination of ingredients in the protective coating of the probe to extend the term of oxidation resistance.
- c. Determine the best combination of compensated lead wires for use with immersion type probes.
- d. Incorporate latest state-of-the-art materials as potting and sealing elements in the base of the probe.

1.1 Statement of Work Cont....

- e. Determine effects of reactions between oxide coatings and tungsten in relation to the emf output.
- f. Establishment of rates of erosion for different types of refractory coatings such as tungsten disilicide, carbides and cermets when subjected to high velocity, high temperature gas streams.

1.2 Progress

Accomplished during the current reporting period were:

- a. Visit to NASA, Huntsville, on 24 July 1963 for conferences with Mr. Harlan Burke and Mr. William Borden of M-ASTR-IM, and conference with Mr. W. A. Record of M-P & C-MPA.
- b. During the conferences with M-ASTR-IM personnel, a design approach for the first three gauges to be delivered was selected. In these gauges, the general design used in ACL Probes, Type 4734, will be incorporated. Features are as follows:
 - (1.) Immersion depth will be two inches.
 - (2.) Diameter at the greatest cross sectional measurement of the immersed portion of the gauge will be not larger than that dimension which will be accommodated by a 7/16" - 20 UNF male fitting.
 - (3.) Mounting will be made by means of a 7/16" - 20 UNF female boss, or screwed connection.
 - (4.) Geometry of the immersed portion of the gauge will be either: tapered rhombus, tapered biconvex, or tapered cylinder. The final geometry selection will be based upon results of analysis and test of such shapes for their resistance to bending, shear, vibration, and shock. Subsequent tests for response (under both no-flow and flow) may also influence the final selection.

1.2 Progress Cont....

- c. Literature search for high temperature electrical insulators has not thus far yielded a readily available insulator for use at temperatures greater than 2480°C.
- d. Results of an independent calibration of a group of ACL Type 4734 gauges were made available to ACL by M-ASTR-IM personnel, and were examined critically. Results of this examination are presented in Section III of this result.
- e. A minimal investigative program, directed toward the possibility of constructing a gauge incorporating no insulation in the immersed portion of the gauge was presented verbally to M-ASTR-IM personnel. It was decided that this approach to the high temperature insulation problem be pursued on a modest basis. Details are given in Section III of this report.
- f. Two of the ACL Type 4734 gauges, previously subjected to tests to failure in a 1/16 scale version of a rocket engine exhaust by NASA, were forwarded to ACL for "post mortem" examination and failure analysis. Results of this examination will be presented in the next monthly progress report.
- g. Requests were made by ACL for the following:
 - (1.) A print of the high-speed cinematography taken during the tests mentioned in Para. f. above for study and analysis.
 - (2.) Approval for acquisition of a plasma spray gun used on a previous program as GFE.
 - (3.) Approval for acquisition of a gaseous hydrogen regulator for use in a high temperature test setup at ACL, either as GFE, if available, or by purchase.
 - (4.) Approval for acquisition of a suitable optical pyrometer for use in high temperature tests, and calibrations at ACL.

1.2 Progress Cont....

- (5.) NASA assistance in obtaining a translation of a Russian report entitled "Plasticheskiye Massy", 1963, No. 4, pp 37-39, concerning an adhesive designated as "VK-6". Information received at ACL indicates that these materials are useable to 1000°C, being based on organosilicone resins filled with fibrous asbestos.
- (6.) Identification by serial number of the ACL Type 4734 gauges tested at NASA, and at Southern Research Institute. This identification is required to enable ACL to obtain the greatest amount of information from analysis of the results of the tests.

SECTION II

PAST PROGRESS

2.0 General

The first reporting period, 17 June 1963 to 1 July 1963, was covered in ACL Progress Report No. T-1097-1, delivered to NASA on 15 July 1963.

2.1 Form

An analysis was made of a set of hypothetical flow conditions delineating the mach regime in which the gauges might be required to operate, if mounted in a high velocity, high temperature flow field. The mounting and performance of a gauge in this, or a similar field, as regards response, oxidation, erosion, shock, vibration and dynamic loading, is important to the program.

2.2 Coatings

Various types of oxidation and erosion resistant coatings were discussed, with a resulting conclusion that the diffused coatings exhibited the greater advantages in this requirement.

2.3 Electrical Insulation

Samarium oxide was examined for possible use, but was rejected because of a low melting point.

2.4 Errata

In Figure No. 1, of Report T-1097-1, the scale for Mach number was inadvertently omitted. A corrected Figure 1 is included at the end of this report.

SECTION III

CURRENT PROGRESS

3.0 General

The current reporting period was devoted principally to resolution of details pertinent to fabrication of the first set of three gauges scheduled for delivery on 17 October 1963. These early models will be intended for design proofing tests in the 4000°F - 4500°F temperature range.

Concurrently with the above, materials research was continued and preparations were made for preliminary testing of components to be used in the project.

3.1 Objectives

3.2 Form

In Report No. T-1097-1, a discussion of factors relevant to the conditions of mounting under which the gauges are to be used was presented. The discussion included an analysis of the variations in Mach number which might be seen by the gauge, were it to be used in a high mass velocity medium. Also discussed were factors influencing design approach under no-flow, or locally turbulent conditions.

During further discussions with NASA technical personnel, it developed that, at present, flow conditions could not be well defined until a mounting point in the area of ultimate use could be selected. Therefore, it was agreed that first group of gauges to be delivered should incorporate a geometry applicable to the widest possible variety of useage consistent with obtaining test data within a limited set of early objectives.

3.2 Form Cont....

The geometry of the gauges is defined as follows:

- a. Immersion depth - approximately 2 inches.
- b. Mounting - screwed, male, 7/16" -20 UNF.
- c. Sheath diameter - minimum .250 inches, maximum any dimension acceptable by the 7/16 -20 fitting.
- d. Sheath form - tapered cylindrical.
- e. Base configuration - similar to ACL Type 4734 gauges, but incorporating improvements in materials, design, and potting.

The design approach given above in Paragraphs 3.2, a. through e. are discussed in more detail below.

3.2.1 Immersion Depth

The immersion depth of 2" was chosen for the following reasons:

- a. Calibration tests performed by an independent agency (Southern Research Institute) see Figures 1, and 2, revealed a consistency of deviation from actual temperature with immersion depth. The tests were limited to 1-5/16" because of the dimensions of the isothermal cavity used. Extrapolation of the test data of temperature versus immersion depth yields an immersion depth of approximately 2" for stabilization of the predicted output emf curve vs temperature with a tolerable deviation from actual temperature at the sensing junction of the gauge.
- b. The two inch immersion seems a reasonable cantilever under the requirements for shock and vibration. Since finite values for strength of the materials is not presently available, no attempt at a rigorous structural analysis of the sheath is being made at this time. Others* are conducting such a test program, however, and results are expected to be available before the end of this program. Meanwhile, ACL is planning vibration and shock tests of prototype gauge assemblies.

3.2.2 Mounting

Mounting provisions are currently predicated upon available and existing means of insertion for test and evaluation; i.e., a 7/16" -20 UNF boss.

3.2.3 Sheath Diameter

Maximum sheath diameter is presently limited by the 7/16 -20 screwed connection.

3.2.4 Sheath Form

A tapered cylindrical form was selected for the first group of probes as a suitable compromise between desired shape for low drag at high mass velocity flow, because of decreased projected area, decrease of mass from base to tip for resistance to dynamic forces, vibration and shock, improved response because of decreased mass at the sensing junction. A practical consideration also influencing the selection of this shape was that of cost. The tapered rhombus, wedge, and biconvex cross sectional shapes all would be considerably more difficult to produce and would, as a consequence, cost more per unit. It is felt, therefore, that the overall objectives of the program would be best served by conserving funds wherever possible, while performing investigations and collecting the largest practical amount of test data for analysis.

3.2.5 Base Configuration

The ACL Type 4734 gauges incorporated a base configuration which was shown by test to be unsuitable for the following reasons:

- a. The temperatures reached at the "cold" end of the sheath were considerably above those anticipated. Because of the high temperature rise in the sheath of the gauge, the body metal (series 304 stainless steel) was raised to temperatures higher than desirable, even during relatively short runs, despite the presence of a large heat sink in the steel deflection shield.

3.2.5 Base Configuration Cont....

- b. The rise in temperature in the body caused the potting material (Sauereisen No. 25) to swell and, in some cases, run and extrude through openings.
- c. In one instance, a gauge sheath was observed to have been broken at its juncture with the deflection shield after a run under high temperature and high mass velocity. The exact mechanics of failure are not yet known. It is suspected that vibratory contact of the tungsten sheath against the base, across a void created by loss of the Sauereisen may have been contributory, because of loss of damping.

3.3 Prototype Design

The design features of the prototype gauge sheath, and forming mandrel are shown in Figure 3, ACL Drawing Number SK4735-03. The block of Drawing Numbers 47XX-XX has been assigned to this project in order to properly identify all gauges, components, piece parts; etc. It is emphasized that the features described are "first look" and may be subject to modification prior to fabrication.

3.3.1 Sheath Formation

The sheath and junction of the ACL Type 4735 gauge is formed by a vapor deposition process. This work is performed under a process developed by San Fernando Laboratories, Inc., Picoima, California. The sheath assemblies are made in accordance with drawings provided by ACL.

Steps in the fabrication are as follows:

- a. A mild steel mandrel is machined to the interior configuration desired, with allowance made for the electrical insulator, differences in expansion coefficients, etc.
- b. The mandrel is provided with a steel mounting base for securing to the forming apparatus.

3.3.1 Sheath Formation Cont....

- c. A piece of Tungsten - 26% Rhenium (W-26 Re) wire of the desired diameter, and length, is introduced through a hole in the tip of the mandrel, and is secured with a set screw in the mandrel holder. The protrusion of the W-26Re wire is set to the required distance outside the mandrel tip. It is important that the Tungsten-Rhenium alloy wire does not extend through the Tungsten, such that it is exposed after fabrication, to avoid the formation of an eutectic during the oxidation resistant coating process.
- d. The mandrel assembly is located in a closed furnace, and is heated electrically to a predetermined temperature. A controlled flow of Tungsten Hexafluoride (WF_6) is introduced into the furnace and the Tungsten is deposited on the mandrel to the required thickness. In the process, the Tungsten conforms precisely with the form of the mandrel.
- e. The junction between the Tungsten sheath and the Tungsten - 26% Rhenium alloy wire is formed simultaneously with the sheath formation described in d. above.
- f. The mandrel may be removed from the deposited assembly by either withdrawing the mandrel after heating, or, if the shape of the mandrel precludes withdrawal, by etching the mandrel out with Hydrochloric acid (HCl) which does not attack either the Tungsten or the Tungsten-Rhenium. Minor surface finishing is accomplished by grinding. Trimming is performed with an air-driven, high speed slotting disc, surfaced with diamond or carbide particles.

3.3.2 Base Fabrication

It is contemplated that the base of the gauge will be comprised of the following elements:

a. Shell

The shell will be formed by machining stainless steel to the desired configuration. Because of difficulties encountered in previous high temperature tests, it was evident that the

3.3.2 Base Fabrication Cont....

a. Shell Cont....

base must be protected from the high temperatures created by conduction. Therefore, it is intended to employ capacitor grade tantalum in the portion of the gauge immediately in contact with the sheath. The tantalum, in turn, would be protected with an intumescent coating such as Dynatherm D-65*, which protects against both oxidation and high velocity. Design of the base of the gauge is proceeding. Drawings will be included in the next report.

3.3.3 Base Insulation

Since base temperatures, although relatively high, are less than those to be seen at the tip of the gauge, an electrical insulator with the capability of remaining an electrical insulator at temperatures less than the maximum temperature at the tip, can be employed. A suitable insulator for this purpose is Magnesium Oxide (MgO). This material offers the following characteristics:**

Melting Point - 4800 - 5070°F

Coef. Thermal Exp. @ 2200°F - Approximately $7.7 \times 10^{-6}/^{\circ}\text{F}$

Density - 2.5 - 2.6 gm/cc

Resistivity - ohm/cm @ 2500°F - 10.5×10^3

Thermal Shock Res. - Fair

Hardness - 5 Mohs scale

It is planned to use this material in the finely divided form, and compact it mechanically to attain sufficient rigidity to prevent creation of voids in the base cavity. Use of the material in the powder form may also provide internal damping in an attempt to avoid vibration damage.

3.3.4 Lead Wires

In the ACL Type 4734 gauges, Minneapolis-Honeywell compensated lead wire was used, principally because of its availability in meeting delivery schedules.

Several other types of compensated lead wire have since become available from sources as follows:

Thermoelectric, Saddlebrook, N. J.
Tuttle & Kifft, Chicago, Illinois
Hoskins Manufacturing Company, Chicago, Illinois
Harco Laboratories, New Haven, Connecticut

Samples of these materials have been ordered for evaluation. They will be tested for their characteristics, and the best type will be used in the ACL gauges.

Previously, the Tungsten-Rhenium lead wire was directly brought out to a transition. Attachment of the Tungsten compensated lead wire to the Tungsten sheath was made by brazing. It is presently planned to join an extension of Tungsten to the Tungsten sheath by vapor deposition of pure Tungsten. It is further planned to enclose the selected compensated lead wires in a swaged stainless steel assembly, insulated with MgO. The length of the lead wires will be four feet, unless ACL is advised by NASA that some other length would be more desirable.

3.3.5 High Temperature Insulators

In searching for electrical insulation adequate for use at elevated temperatures it has been found that ordinarily available literature contains little or no information regarding electrical characteristics at temperatures above approximately 1600°C (2912°F). Contact has been established with Hughes Aircraft Company, Documentation Research Section, Culver City, California, which is engaged in Electro-Physical Properties of Matter research under a Government contract. A similar contact has been established with Purdue University, Thermophysical Properties Research Center. Results of inquiries to these organizations will be included in subsequent reports.

3.3.5 High Temperature Insulators Cont....

In parallel with the literature searches mentioned above, ACL has compiled empirical data, as well as comparisons of existing published data concerning high temperature insulators. A comparison of values is tabulated below.

TABLE I

OXIDE INSULATORS, PROPERTIES

<u>Property</u>	<u>BeO</u> <u>Beryllia</u>	<u>Al₂O₃</u> <u>Alumina</u>	<u>ZrO₂</u> <u>Zirconia</u>	<u>MgO</u> <u>Magnesia</u>	<u>ThO₂</u> <u>Thoria</u>
Melting Point °F	4650	3700	4830	5070	5970
Hardness (Mohs)	9	9	6.5	6	6.5
Density gm/cc	3.0	3.97	6.1	3.6	9.7
Thermal Conductivity (cal/sec °C-cm ² /cm					
100°C	.525	.072	.005	.086	.025
600°C	.112	.022	.005	.028	.010
1000°C	.049	.015	.006	.017	.007
Thermal Shock Res.	Excellent	Good	Fair	Fair	Fair
Electrical Resistivity					
ohm/cm 100°C	-	1 x 10 ¹⁵	1 x 10 ⁶	-	-
600°C	-	2 x 10 ¹⁰	8 x 10 ³	-	1 x 10 ⁷
1000°C	8 x 10 ¹²	2 x 10 ⁷	1 x 10 ²	1 x 10 ¹³	1 x 10 ⁵
1200°C	3 x 10 ¹²	2 x 10 ⁵	-	8 x 10 ¹¹	2 x 10 ⁴
1600°C	2 x 10 ¹⁰	-	-	-	becomes conductor
2200°C				exp. upper*	
2450°C	exp. upper limit*			limit	

* Exact value not known

An examination of Table I shows that Alumina is eliminated because of its low melting point, Thoria because of its poor thermal shock characteristic and low resistivity, Zirconia because of its low resistivity.

3.3.5 High Temperature Insulators Cont....

Of the two materials remaining, Beryllia is clearly the better, because of its characteristic of remaining an electrical insulator to a higher temperature than Magnesia, even though the Magnesia has a higher melting point.

Another family of materials exhibiting high melting points, but concerning which little information is available electrically, is the Zirconates. Table II, below, lists those with high temperature characteristics in the range of interest.

TABLE II

ZIRCONATES*

<u>Material</u>	<u>Melting Point °C</u>
Ba·ZrO ₂	2700
3Be · 2ZrO ₂	2535
Ca · ZrO ₂	2345
MgO · ZrO ₂	2120
SrO · ZrO ₂	2700
ThO ₂ · ZrO ₂	2800
ZrO ₂ · SiO ₂	2420

Although it is obvious from Table I that Zirconium Oxide, which is common to all these compounds, is unsuitable as an insulator in its pure form, the degree to which the combination of compounds affects their electrical characteristics is not known. The basic measurement of resistivity over a wide temperature range is beyond the monetary scope of this project. However, it is felt a limited investigation should be performed to determine whether there may be grounds for future work on these materials. They will also be examined for possible use as protective materials

3.3.6 Protective Coatings

Protective coatings, as applicable to this program, are classified in two groups, oxidation resistant, and erosion resistant. Many such coatings have been developed over the past several years, and a great amount of data concerning their characteristics has been accumulated. Unfortunately, these data are extremely limited at temperatures above 3000°F. The secondary effects of the coatings upon the substrate is also an area in which little information is thus far available. The general thermal protective mechanisms upon which the coatings are based may be described as follows:

3.3.6.1 Ablation

The most common types of ablative coatings employ polymeric binders which undergo thermal decomposition to produce a firm porous carbon char, as well as gaseous by-products. The process is endothermic (heat absorbing), thus protecting the substrate against heat. The effectiveness of the process is measured by the amount of heat absorbed per unit weight of protective material used. This property varies, not only with the thermal conductivity of the material and the temperature, but also with other more subtle mechanisms working simultaneously.

3.3.6.2 Sublimation

As is seen in the definition of this latent process, subliming materials neither melt nor decompose, but endure a change of state directly from the solid to the gaseous form. Large quantities of heat are thus absorbed until the subliming material is used up, at which time the protective mechanism ceases. Thus, if the heat of sublimation, the temperature and the quantity of the material, are known, the length of time for protection can be predicted.

3.3.6.3 Decomposition and Dehydration

When inorganic compounds decompose, appreciable amounts of heat are absorbed. Hydrated salts require large amounts of heat as the

3.3.6.3 Decomposition and Dehydration Cont....

water molecules are forced out of the salts by the heat of hydration. Water itself is an outstanding example of a heat absorber as it changes from the liquid to the vapor state by the latent heat of vaporization.

3.3.6.4 Insulation

The efficient insulator protects from the effects of temperature by the introduction of a high thermal resistance (low thermal conductivity) between the substrate and the high temperature environment. Even the best available insulator, however, can not protect the substrate forever because of a finite thermal capacity in the substrate. An exception might be in the case where the substrate is capable of releasing the heat it has taken up by radiation, conduction, or convection, at a rate consistent with an acceptable temperature rise.

3.3.6.5 Intumescence

Intumescence occurs when a protective material swells and foams, upon exposure to high temperature, thus producing a small celled mass, whose coefficient of thermal conductivity is small. Additionally, the exposed surface chars to afford erosion resistance. These materials have measured characteristics as follows:

TABLE III

INTUMESCENT COATINGS

<u>Exposure Temperature</u>	<u>Medium</u>	<u>Coating Thickness</u>	<u>Heat Flux</u>	<u>Time</u>	<u>Matl.</u>
2000°F - 3000°F	LOX-Fuel	.250 inch	Not Known	30 min.	Rigid Poly Urethane
**2000°F	LOX-Kerosene	.125 inch	Not Known		"
350°F - 6300°F	Not Known	.300 inch	1400 Btu/ft ² /sec	10 min.	Polymer
5000°F	Not Known	Approx. .020 inch	Not Known	60 sec.	Polymer
*2000°F - 3000°F	LOX-Kerosene	.090 - .125 inch		150 sec.	Urethane

3.3.6.5 Intumescence Cont....

The family of intumescent materials may incorporate several of the protective mechanisms described previously. Materials such as phenolics, ester resins, phenol-form, aldehydes, polyesters, silicones, and urethanes, may be used.

In addition to their flame resistant characteristics, these materials may also be compounded to resist the deleterious effects of various oxidizers, such as N_2O_4 , LOX, IRFNA and fuels such as Kerosene, UDMH, H_2 .

They are an interesting family of materials, and it is felt that a rather hard look should be taken at them for a possible application in this project.

3.3.7 Diffused Protective Coatings

The ACL Type 4734 gauges, produced under NASA Contract NAS 8-4548, were provided with a diffused protective coating on the sheath. The materials in the coating were of two general groups: Disilicides, applied with a hot pack process (*Durak-B, Durak MG) and Silicones, applied by vapor deposition.

A large body of data, involving several thousands of hours of test, is available from Pratt and Whitney, Ling-Temco-Vought, Avco, and others, regarding the disilicides of Molybdenum and Tungsten, but only to temperatures in the order of $3000^{\circ}F$, in oxidizing media.

Early tests performed at NASA, Huntsville, on a few of the Type 4734 gauges, were productive, in that the gauges operated successfully for short periods of time. Two of the gauges, thus tested, were transmitted to ACL on 25 July 1963, and are being examined currently. Results of this analysis will be presented in a future report. In general, it can be said, on the basis of a first look, that the protective coatings held up quite well.

To aid in the analysis of this critical phase of the project, it is requested that a print of the high speed color films, taken during the tests, be made available to ACL at the earliest possible time.

3.3.7 Diffused Protective Coatings Cont....

Tests previously performed at ACL, in a highly oxidizing medium at temperatures from ambient to approximately 4000°F revealed that the disilicide coating protected the gauges for a total of 19 minutes, during six cycles, and for 2 minutes at near 6000°F in a reducing medium for one cycle.

3.4 Test Program

3.4.1 Oxidation Resistance

Presently in fabrication are six tungsten test thimbles for use in a series of oxidation resistance tests. These parts are formed by vapor deposition, and will be essentially identical in composition to the ACL Type 4735 prototype gauges. Dimensions of the test articles will be .250 nominal O.D. by 3.0 inches in length. Wall thickness will range from .010 inches to .060 inches. The high temperature duct for this series of tests is shown in Figure 4.

3.4.2 No Insulation Test

During a previous high temperature instrumentation program conducted by ACL, a Tungsten vs Tungsten-26% Rhenium thermocouple was used to measure temperatures in the order of 5000°F in an induction furnace. Good correlation was obtained between the predicted output curve for the couple, and measurements taken with optical pyrometry (corrected for emissivity). The atmosphere ranged from vacuum to reducing.

In this setup, the thermocouple was stretched across the high temperature area, with the junction located in the "hot" zone. The wires were 180° apart. No insulation, other than ceramic feed-throughs in the relatively cool walls of the furnace, was used.

In the current investigation, it is desired to establish the relationship between emf output of a thermocouple and angularity between the wires in the pair. It is therefore planned to conduct this test with iron vs constantan pairs, to establish whether there is some

3.4.2 No Insulation Test Cont....

minimum angle beyond which accuracy is outside tolerable limits. Control of test parameters, with iron-constantan, will be more practical at lower temperatures, as well as cost.

In common practice, a thermocouple is fabricated with a junction between two wires, an electrical insulator between the wires and a supporting sheath. The tip is not insulated, but has a finite spacing between the wires. The electrical insulation is provided to minimize current flow between wires and preclude averaging of output. Otherwise, the temperature inferred from the emf output of the couple would be the average temperature rather than the temperature at the junction. However, any losses in accuracy due to the spacing at the tip, if present, are not appreciable enough to cause a deviation in output beyond ordinary limits of tolerance.

It is planned, therefore, to set up the test to run at carefully controlled temperatures, with angularities from 180° to some lower limit determined by test. At the same time, the temperature at the junction of the test thermocouple will be monitored with a calibrated standard probe.

Should this approach show merit, the tests would be repeated with temperature limits increased, with the end objective of fabricating a gauge incorporating no electrical insulation within the sheath. Results of these investigations will be presented in future reports.

3.4.3 Other Refractory Sheaths

Although limited success has been experienced with Tungsten as a sheath material for the ACL Type 4734 gauges, investigations into other refractory materials has continued. Results of the most recent investigations are as follows:

3.4.3.1 Tungsten-Rhenium Alloys

Tungsten-Rhenium alloys had been considered for use as an outer sheath because of their greater ductility, as compared to pure

3.4.3.1 Tungsten-Rhenium Alloys Cont....

Tungsten. Others* had reported success with this technique, as a means of fabricating high temperature thermocouples. However, when an attempt was made to apply a protective disilicide coating, an eutectic formed. Therefore, since protection is required and alternate types of suitable protective coatings are not presently available, this type of material has been at least temporarily shelved.

3.4.3.2 Tantalum w/BeO Insulator

Tantalum has been widely used as sheathing because of its excellent formability and high melting point. It had been considered for such use in high temperature thermocouples, until it was reported** that a definite reaction with BeO, after long term exposure at 1600°F, was observed. When it is considered that the speed of reaction increases almost exponentially at the higher temperatures, this combination of materials was dropped.

3.4.3.3 Filament Wound-Tungsten Tubing

Presently under development*** is a new type of structure for Tungsten tubing, in which Tungsten filament approximately .002 inch diameter is angle-wound around a mandrel of the desired i.d., and is laid up in layers to the desired wall thickness. This assembly is then placed in a deposition furnace, and pure Tungsten is deposited to fill the voids between filaments. The end result is a Tungsten tube whose tensile strength may approach that of the filament (300,000 psi). At present, such tubes are a laboratory curiosity. Results of tests on this material will be included in subsequent reports.

3.4.3.4 High Melting Point Materials

A general survey of high melting point materials, from 3000°F to 6800°F has been made, and is presented in Table No. IV. This table has been compiled as a quick reference aid for future research.

*Lachman, Hoskins Mfg. Co.

**Holzel, R., San Fernando Laboratories

***San Fernando Laboratories, Pacoima, California

TABLE IV
HIGH MELTING-POINT MATERIALS*

<u>Materials</u>	<u>Melting Point, °F</u>	<u>Materials</u>	<u>Melting Point, °F</u>
Niobium carbide (NbC)	6800	Molybdenum carbide (Mo ₂ C)	4650
Graphite (C)	6700	Zircon (ZrSiO ₄)	4622
Zirconium carbide (ZrC)	6400	Beryllium oxide (BeO)	4568
Tungsten (W)	6098	Cerium sulphide (CeS)	4440
Titanium nitride (TiN)	5800	Strontium oxide (SrO)	4406
Barium phosphide (Ba ₃ P ₂)	5790	Silicon oxide (SiO)	4406
Titanium carbide (TiC)	5700	Yttrium oxide (Y ₂ O ₃)	4380
Tantalum (Ta)	5440	Niobium (Nb)	4380
Zirconium nitride (ZrN)	5400	Columbium (Cb)	4370
Vanadium carbide (VC)	5090	Vanadium nitride (VN)	4280
Strontium zirconate (SrO·ZrO ₂)	5070	Calcium zirconate (Ca·ZrO ₂)	4230
Magnesium oxide (MgO)	5070	Chromium oxide (Cr ₂ O ₃)	4127
Zirconium oxide (ZrO ₂)	4900	Zirconium silicides (Zr ₃ Si ₂ , Zr ₄ Si ₃ , Zr ₆ Si ₅)	4010-4080
Molybdenum (Mo)	4760	Aluminum nitride (AlN)	4060
Barium zirconate (BaO·ZrO ₂)	4748	Silicon carbide (SiC)	4000
Cerium oxide (CeO ₂)	4712	Barium sulphide (BaS)	4000
Calcium oxide (CaO)	4660	Beryllium nitride (Be ₃ N ₄)	4000

TABLE IV Cont....

<u>Materials</u>	<u>Melting Point, °F</u>	<u>Materials</u>	<u>Melting Point, °F</u>
Barium nitride (Ba_3N_2)	3990	Barium oxide (BaO)	3490
Chromium aluminide (CrAl)	3920	Beryllium oxide-aluminum oxide ($\text{BeO} \cdot \text{Al}_2\text{O}_3$)	3470
Molybdenum aluminide (Mo_3Al)	3900	Silicon nitride (Si_3N_4)	3452
Spinel (MgAl_2O_4)	3874	Chromium carbide (Cr_3C_2)	3440
Titanium dioxide (TiO_2)	3866	Chromium (Cr)	3430
Calcium silicate ($2\text{CaO} \cdot \text{SiO}_2$)	3866	Zirconium (Zr)	3350
Titanium silicide (Ti_5Si_3)	3848	Molybdenum beryllide (MoBe_2)	3344
Beryllium carbide (Be_2C)	3812	Mullite ($3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$)	3290
Aluminum oxide (Al_2O_3)	3722	Zirconium beryllide (ZrBe_9)	3180
Niobium nitride (NbN)	3722	Vanadium (V)	3150
Molybdenum disilicide (MoSi_2)	3686	Silicon dioxide (SiO_2)	3110
Nickel oxide-aluminum oxide ($\text{NiO} \cdot \text{Al}_2\text{O}_3$)	3668	Zirconium disilicide (ZrSi_2)	3092
Beryllium silicate ($2\text{BeO} \cdot \text{SiO}_2$)	3630	Vanadium disilicide (VSi_2)	3020-3180
Barium oxide-aluminum oxide ($\text{BaO} \cdot \text{Al}_2\text{O}_3$)	3630	Molybdenum aluminide (MoAl)	3090
Magnesium sulphide-strontium sulphide (MgS , SrS)	3600	Titanium (Ti)	3074
Nickel oxide (NiO)	3560	Nickel aluminide (NiAl)	3000
Niobium disilicide (NbSi_2)	3542	Zirconium aluminide (ZrAl_2)	3000

3.4.4 Calibration vs Immersion Depth

In an evaluation program* conducted by an independent research institute, several of the ACL Type 4734 gauges were subjected to high temperature calibrations. The number tested and their serial numbers are not known. However, it is assumed the published results are typical of these gauges. In examining the measured output curves, (See Figures 1 and 2) it is apparent they are in fair agreement with calibration curves supplied by ACL, within the temperature range covered.

The curves of output vs immersion depth are of particular interest, in that the influence of immersion depth on deviation from previously calculated and measured value is apparent, for 2000°F, 3000°F, and 4000°F. These were plotted (See Figure 5) as mean deviation from the predicted curve vs immersion depth. Although four immersion depths were examined, only three are of practical use because of the difficulty in reading finite emf values for the 9/16" and 1/2" immersions. A curve can be drawn with only the three points for 1/4", 1/2" and 1-5/16" immersions. However, extrapolation to an exact immersion depth where there is minimum deviation does not seem feasible, because of the uncertainty of predicting the rate at which the deviation slope changes.

It can be shown, however, that in accordance with first law principles, the curve becomes asymptotic with increase of immersion depth, probably approaching 1 mv. mean deviation at about 2" immersion. The parallelism of the curves indicates that an output curve very close to the predicted curve can be obtained with an optimum immersion depth.

It is planned to investigate this area more thoroughly in the second four month phase of this program. Results will be included in future reports.

SECTION IV

PROGRAM FOR NEXT INTERVAL

- 4.0 Objectives for the interval 1 August 1963 to 1 September 1963 will include the following:
- a. Continue literature search for high temperature insulators, and other materials.
 - b. Continue investigations into the "no-insulation" approach.
 - c. Perform "post mortem" examination and analysis of the two ACL Type 4734 gauges previously tested at NASA, Huntsville, in a 1/16 scale rocket engine.
 - d. Set up high temperature test duct and start oxidation rate tests on Tungsten test samples.
 - e. Continue development of base and mounting configuration for first set of three gauges due for delivery 17 October 1963.
 - f. Continue preparations for shock and vibration tests of the prototype gauges.
 - g. Continue analysis of the immersed portion of the gauge with the objective of developing alternate configurations for use, if test results of the prototypes are negative, or if other mounting provisions become available.
 - h. Continue investigations of compensated lead wires.

SECTION VSTATEMENT OF MAN HOURS5.0 Hours By Category

The expenditure of man-hours during the reporting period, and a recapitulation is presented in tabular form below:

<u>Category</u>	<u>Previous Period</u>	<u>Current Period</u>	<u>To Date</u>
Engineering	62.5	101.0	163.5
Clerical	4.0	6.5	10.5
Fabrication	0	14.0	14.0
Conduiting	0	0	0
Drafting	0	2.0	2.0

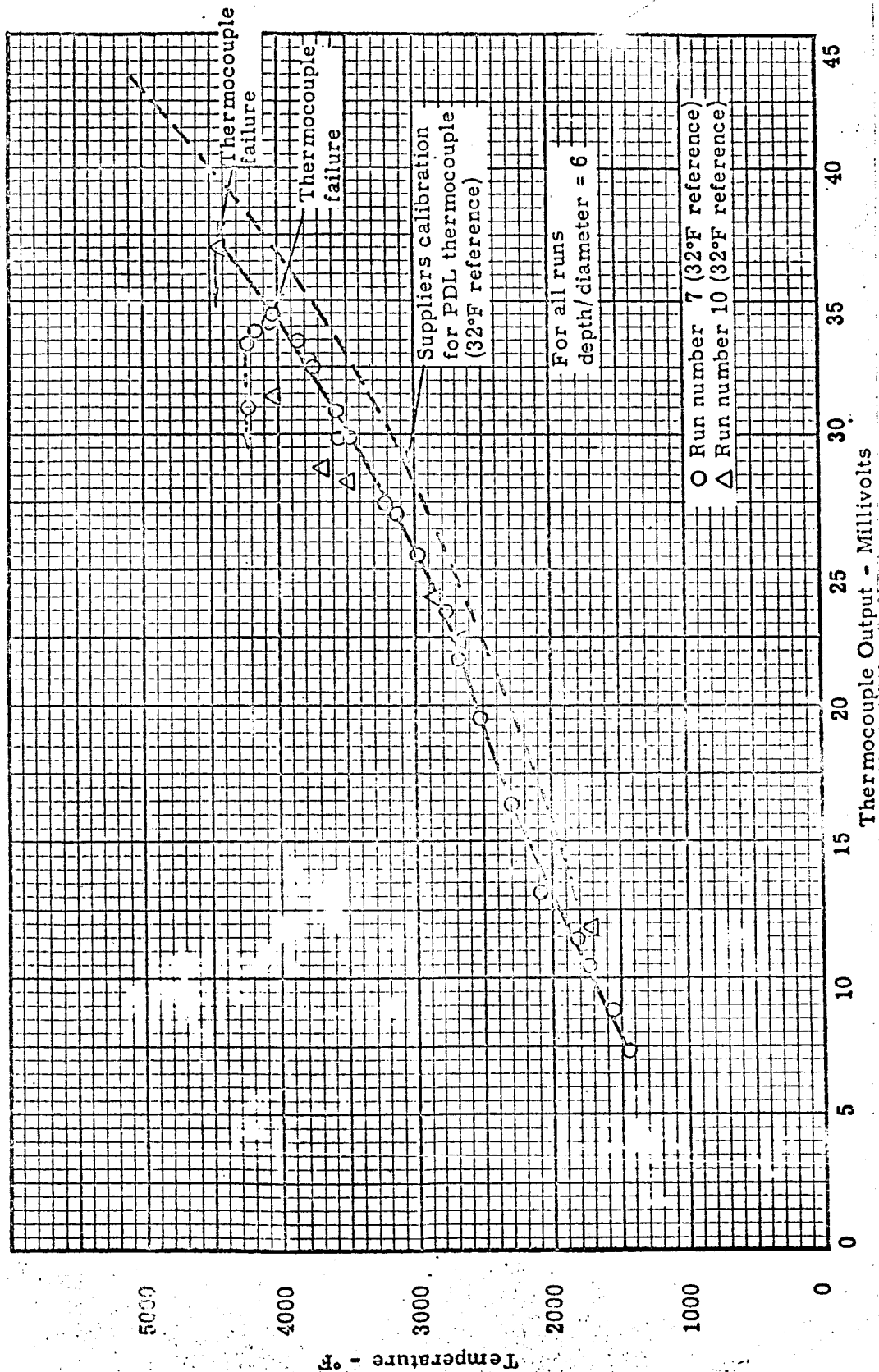


FIGURE 1.

CALIBRATION CURVES

ACL TYPE 4734 THERMOCOUPLE

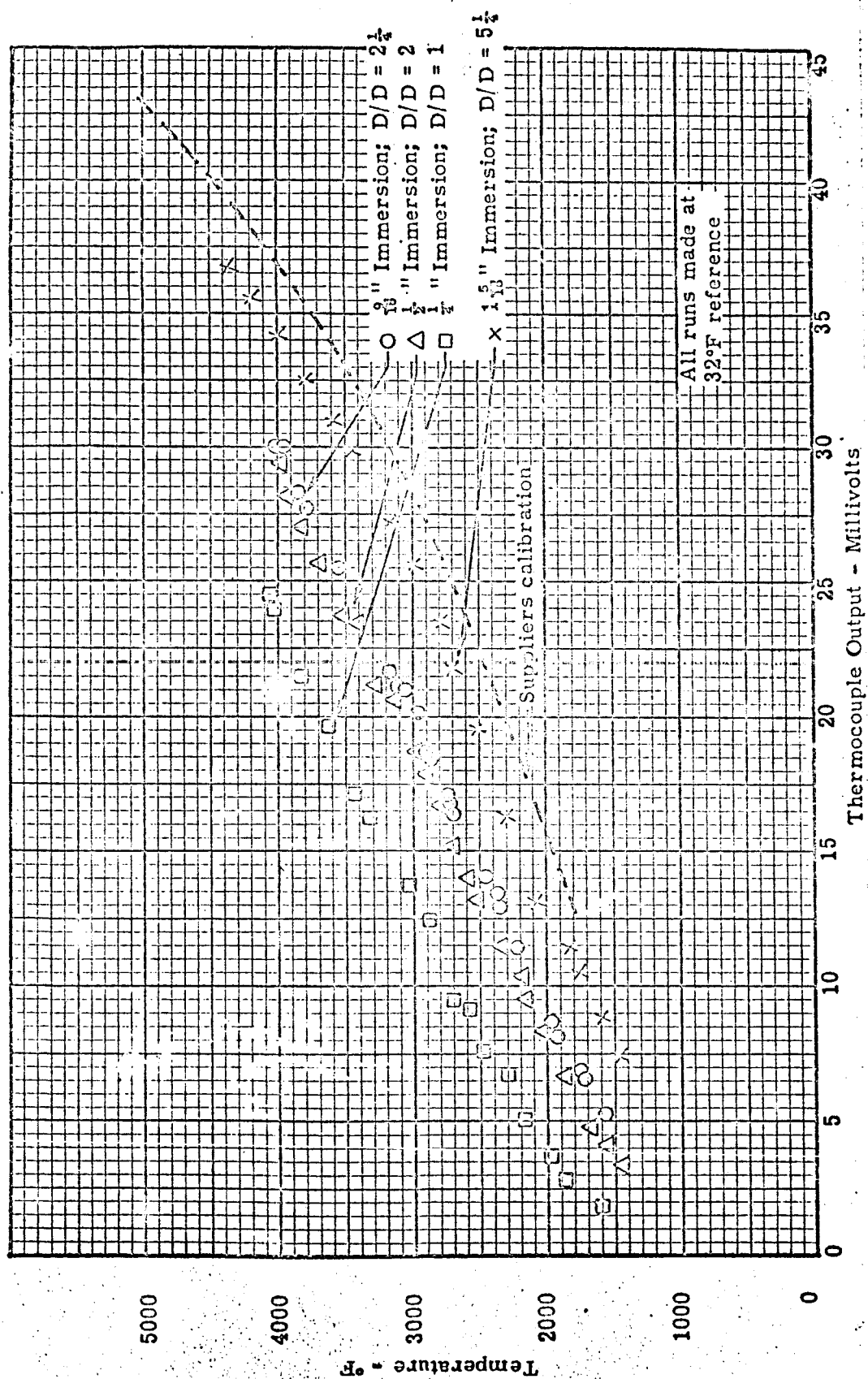


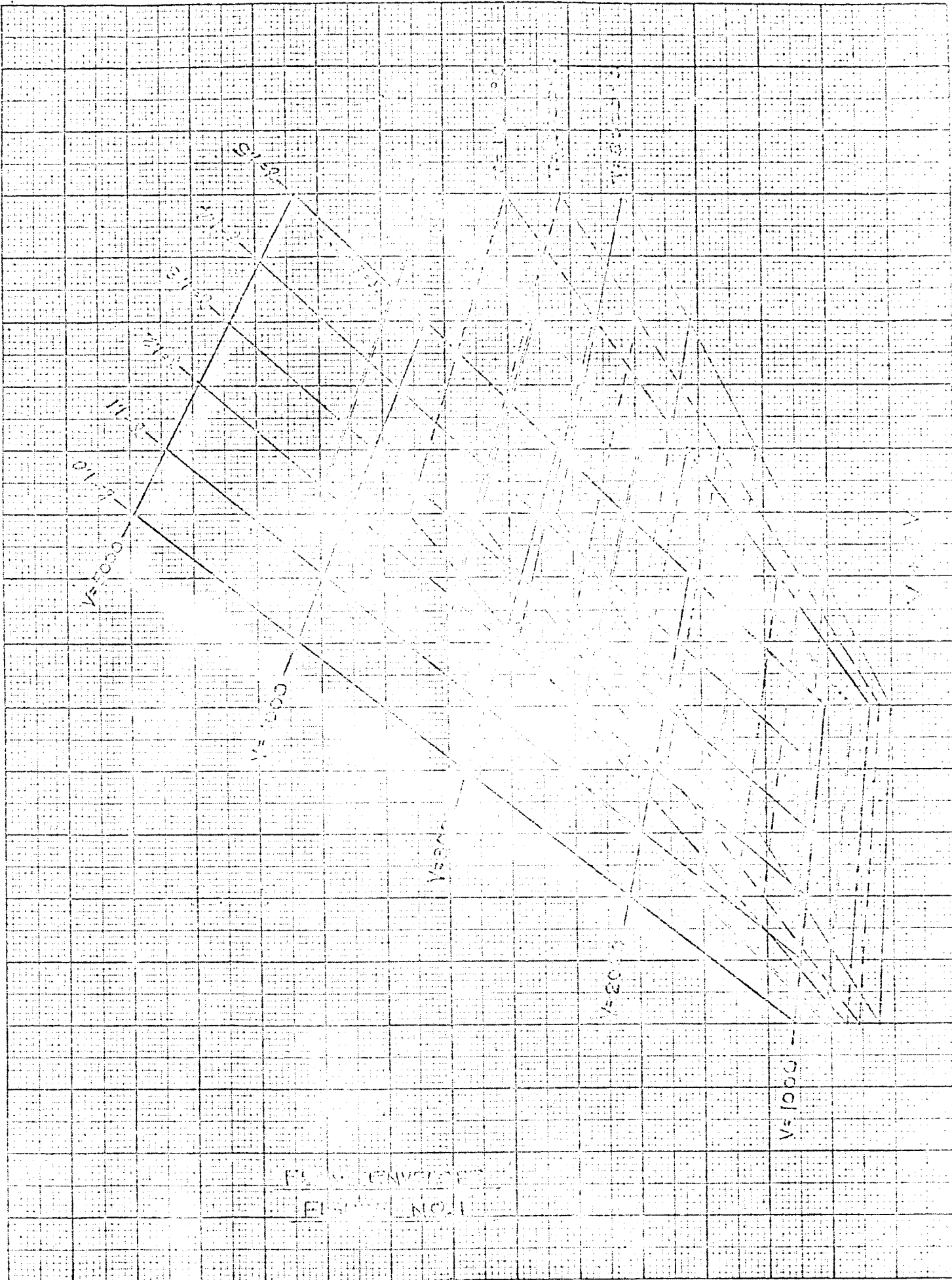
FIGURE 2.
 VARIATION OF ECF OUTPUT WITH IMMERSION DEPTH
 ACL TYPE 4734 THERMOCOUPLE

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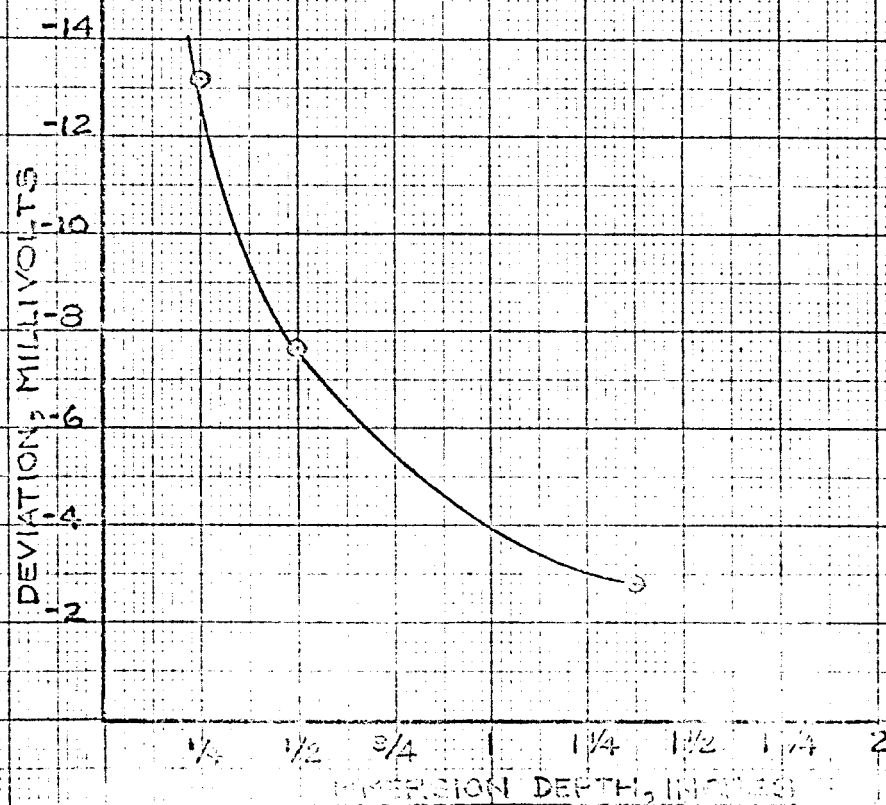
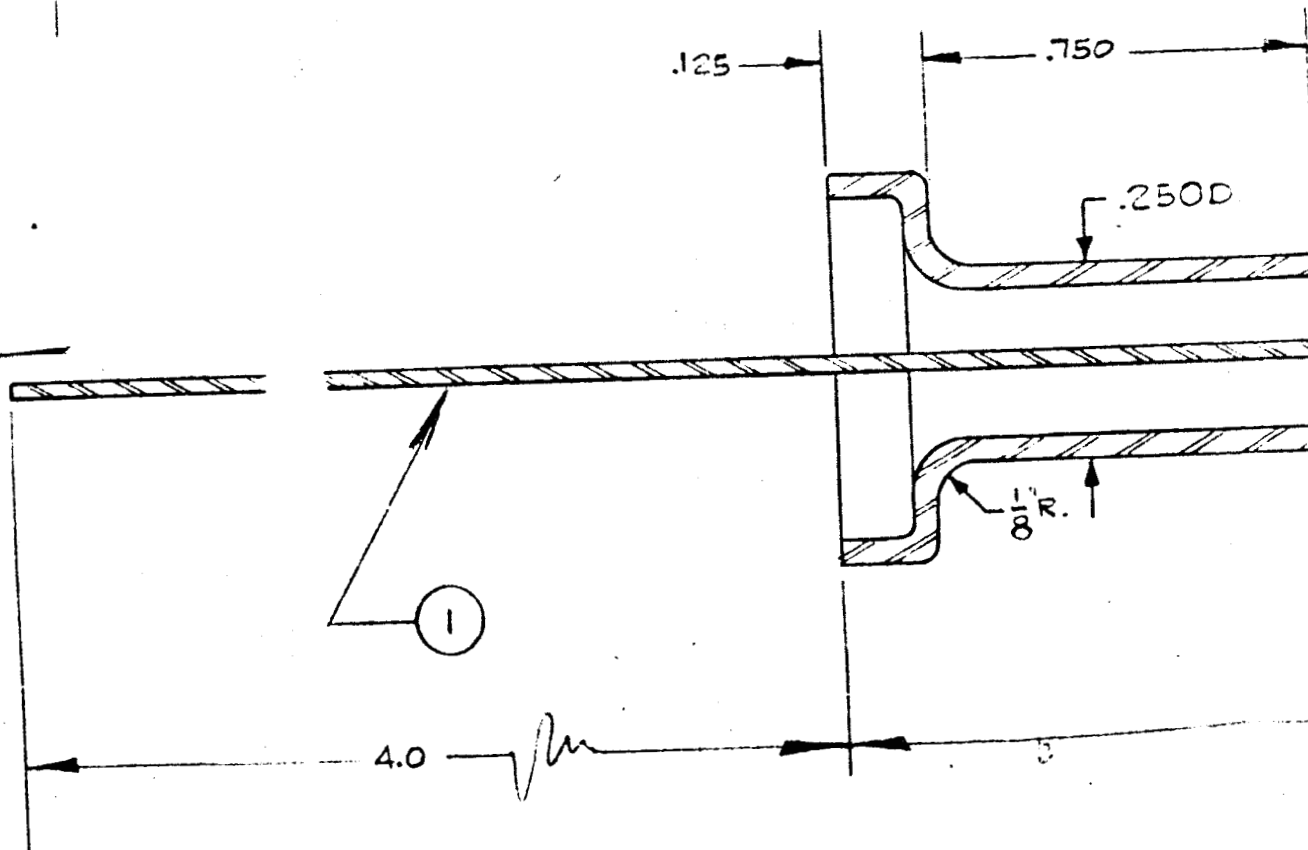
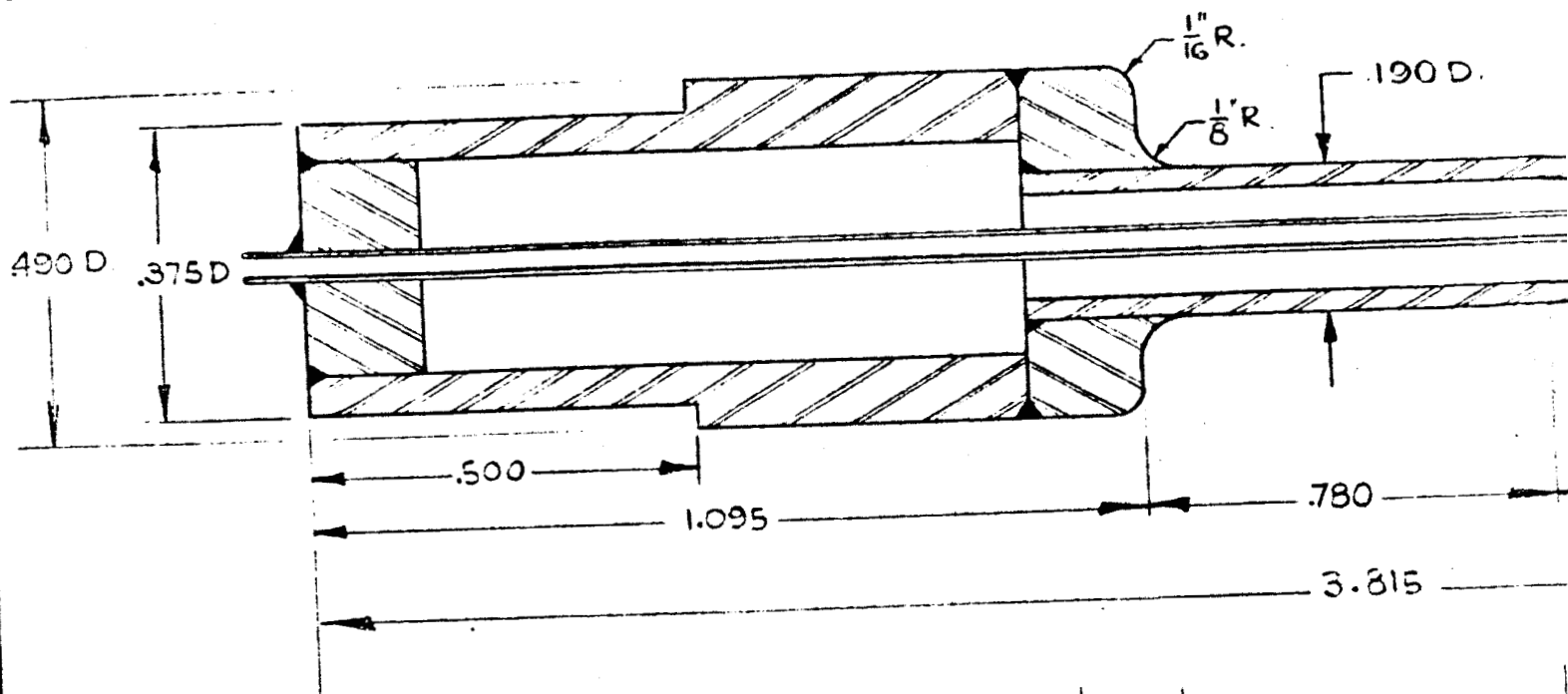
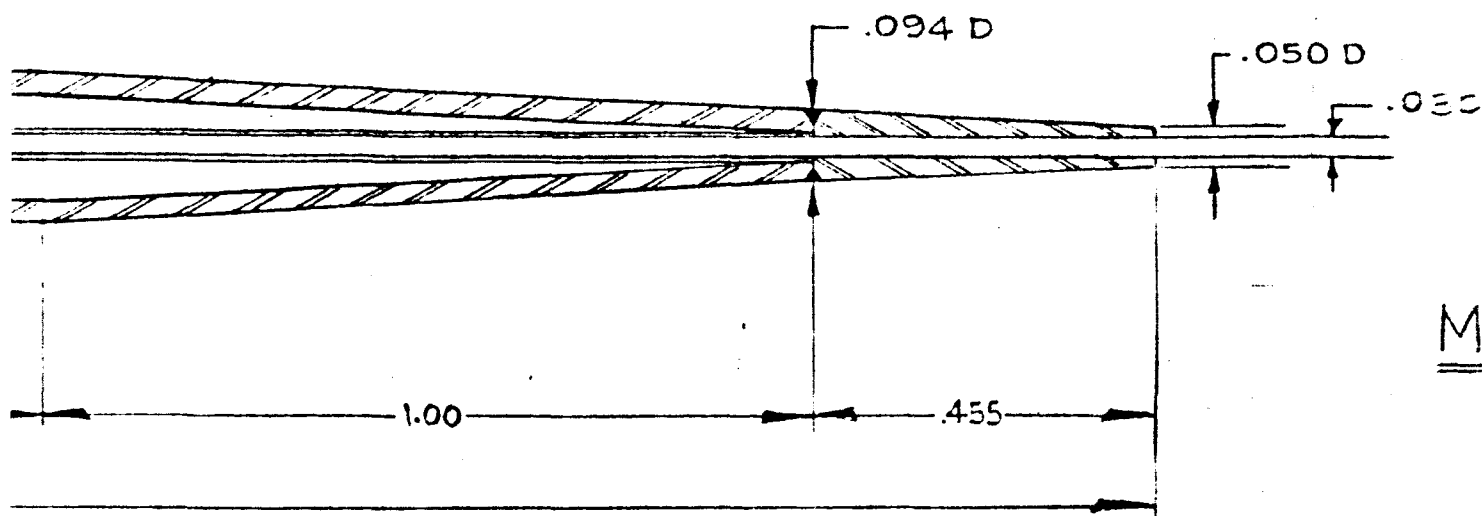
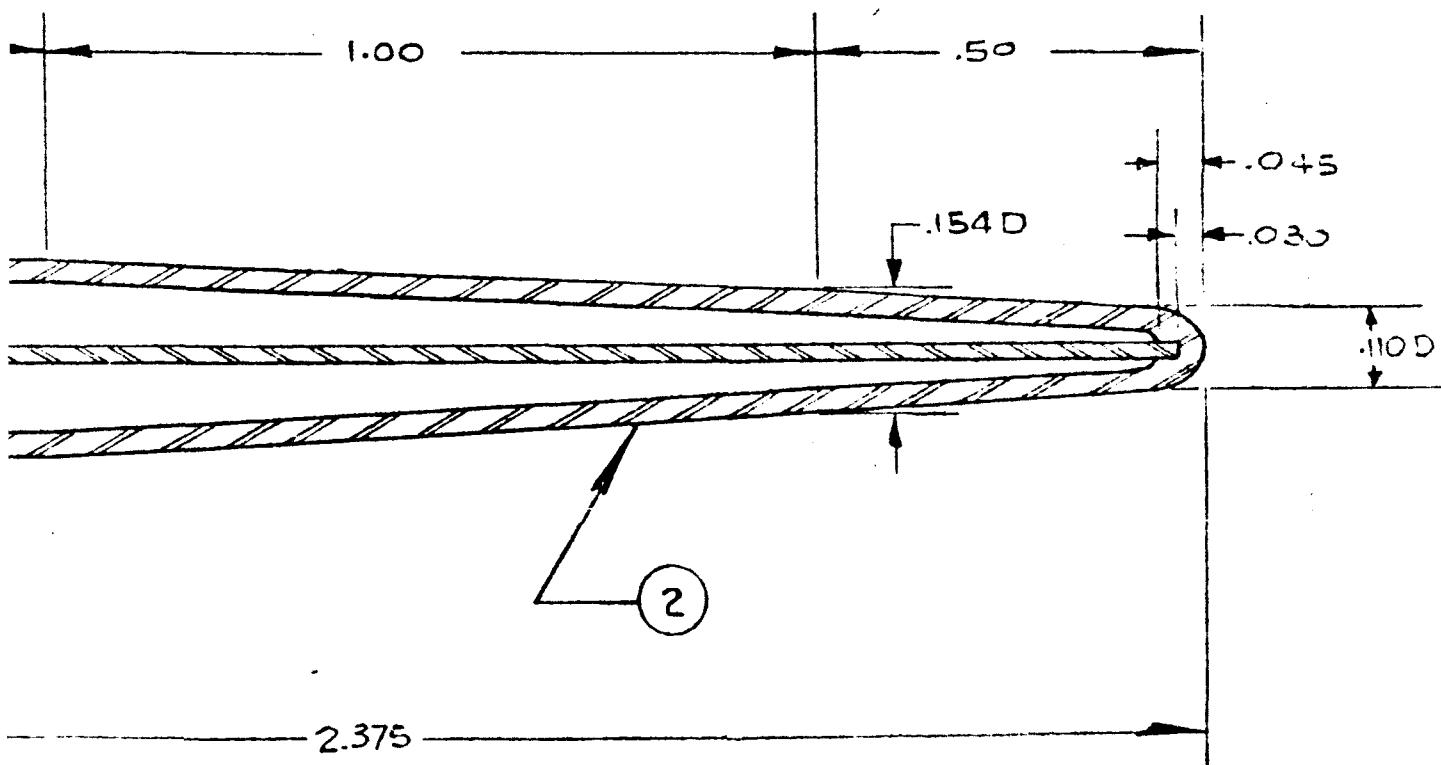


FIGURE 5.
MEAN DEVIATION OF THERMOCOUPLE EMF
OUTPUT WITH IMMERSION DEPTH. ACL
TYPE 4754 THERMOCOUPLE.

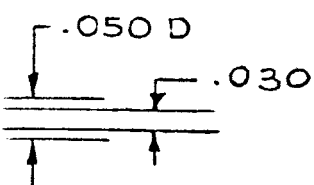




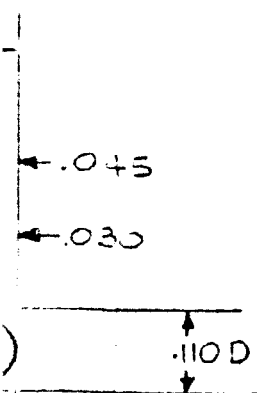
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ITEM NO.	NO. REQ.	DESCRIPTION
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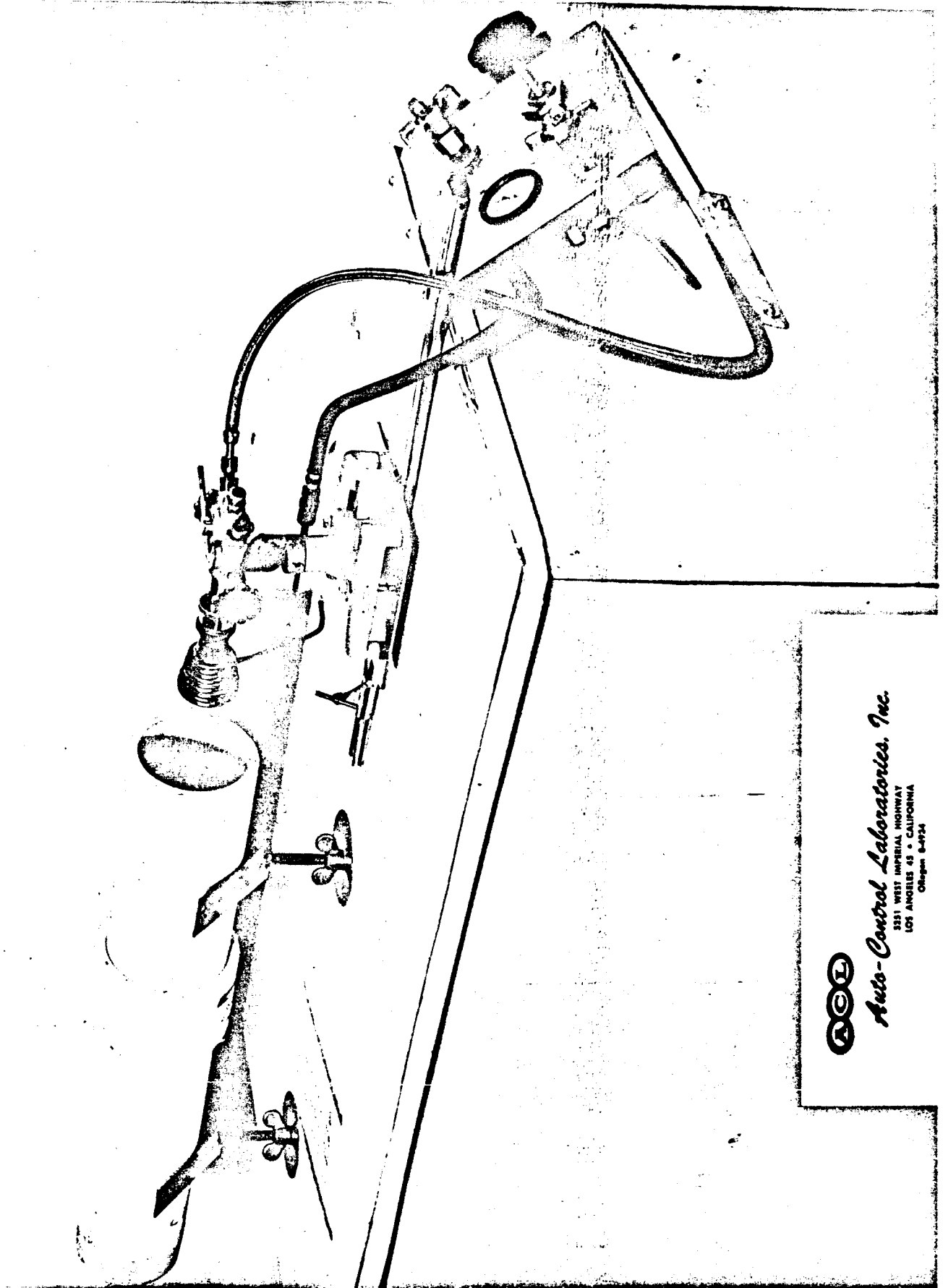


MANDREL, MILD STEEL



SHEATH, W-W26RE

- ① W26RE, 020 DIA.
- ② W, .030 W.T.



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Figure No. 4